REQ. NO. 0-9006F

ULTRASONIC DETECTION OF DEFECTS IN HIGH PERFORMANCE CERAMICS

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Research Contract EMR/DSS 20SU23440-09006F

Annual Report 1981-82

CONTRACT REPORT NO. OSU80-00158

I Introduction

The original and still valid purpose of the investigation is to develop a practical means of NDE for ceramics. The most versatile method was considered to be ultrasonics and research has concentrated on the improvements of existing techniques and the location of those parts of the system requiring further development.

Over the course of the contract period, equipment has been purchased and other equipment has been built, designed or planned. The weakest link in the system has been identified as the transducer and an active transducer development programme is underway. It is proposed to develop an extended focus high frequency immersion transducer.

A start has been made with commercial transducers by detecting flaws in glass and some ceramic superionic conductors. 32 MHz ultrasonics has shown itself a powerful tool. Glass seeds have been analysed down to 50 μm dia and a crack in Hydronium β" Alumina which could not be seen under a powerful microscope was clearly detected by ultrasonics (and later by the SEM).

II Ultrasonics

Although this technique is now well established, the need to find ever smaller flaws, such as the minute fissures in brittle super ceramics, requires more sophisticated instrumentation be developed. Commercial equipment has maximum test frequencies of 25 MHz, but for the small flaws that precipitate failure in high-performance ceramics, frequencies of 100 MHz are necessary. Such require a special pulser-receiver, special transducers, special connections (cables) and special signal processing equipment. Since everything is at the fringes of technical capability, none of the components of the test system were commercially available and have been custom built. The following paragraphs synopsize details of these developments.

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A Pulser-Receiver

Called Spectroscope Model #5030A by Panametrics, this custom built 100 MHz instrument has a gating system allowing only signals in the gate to be analyzed. However, the discrimination is only 35 dB, leaving a residue of the main bang of $\sim$ 0.1 V, i.e., the spectra of all signals below this level cannot be recorded or analyzed. Further improvements in the electronics are presently being contemplated.

B Transducers

Commercial transducers for immersion testing are made with a piezo-electric crystal and a plastic lens. Beyond 25 MHz this construction becomes unsatisfactory because the crystals are too thin and the plastic lenses begin to absorb the signals. Panametrics placed the crystal on a slug of fused quartz allowing high frequency resonance of the very thin crystal but the reflections of the quartz slug (which act as a delay line) interfere with the flaw detection so these transducers are considered useless for our purposes. A program of development of improved transducers is underway.

C Cables

So far, cables have been used between the pulser-receiver and the transducer in the test tank. At higher frequencies, the cable has a distinct influence on the effective frequency of the transducer: e.g., with no cable, the transducer frequency was measured to be $\sim$ 95 MHz, with a short cable 80 MHz and with a 50 cm test cable 60 MHz.

The problem is recognized but as yet no steps towards improvement have been taken.

D Scanner

The focal diameter of -3dB of an ultrasonic transducer of 100 MHz is of the order of 0.1 mm. For 100% coverage of a component, an ultrasonic scanning machine should be able to scan over lines at 0.1 mm intervals with an overlap of 0.02 mm. Although it is hoped that by focus shaping of the transducers this

number can be increased, it is still thought that the machine should have a scanning accuracy of no more than $\sim$ 0.2 mm. The design of such a scanner is currently underway.

III Signal Standardization

The electronic signal from a transducer is the result of integrating the pressures on small elements of area of the transducer. For plane waves, this would simply be the product of the area of the transducer and the sound pressure at any time. Focused transducers have a more complicated sound field as shown by Schlengermann.(1) The single frequency sound field described by the equations used in Schlengermann's paper requires summing over a range of frequencies with varying amplitudes to account for the frequency distribution of the transducer. Treating the surface as a perfect reflector, the pulse-echo technique can be viewed as a transmitter-receiver pair with the transmitter as far below the surface as the receiver is above. By tilting the transducer with respect to the surface, the 'receiver' will sample a different section of the sound field giving rise to a change in the received signal and so a change in frequency spectrum (See Figs. 1 and 2).

For test purposes, the sample is leveled so that the reflection from the bottom remains stationary on the oscilloscope trace. Next, the height of the transducer is adjusted until the signal amplitude is maximized (focal point is on the back surface) then the transducer is tilted until the spectrum of Figure 1 is obtained. This standard is used as it has the smallest low frequency side lobe within the range of adjustment possible with our present equipment. By using "standardized" signals for testing purposes, a uniformity in test results was obtained enabling the definite analysis of the bubble type defects in glass.

IV Glass Defects

While improvements in equipment are made, glass defects have been tested and analyzed. The nominal 25 MHz, F/5 commercial transducer is in fact 32 MHz and this provided the highest test frequencies used to date.

Even though the defects are tagged by a surface marking on the glass, finding them ultrasonically, i.e. matching a 0.2 mm ϕ focus with a < 0.1 mm defect, posed great difficulties until the scanner could search without skipping.

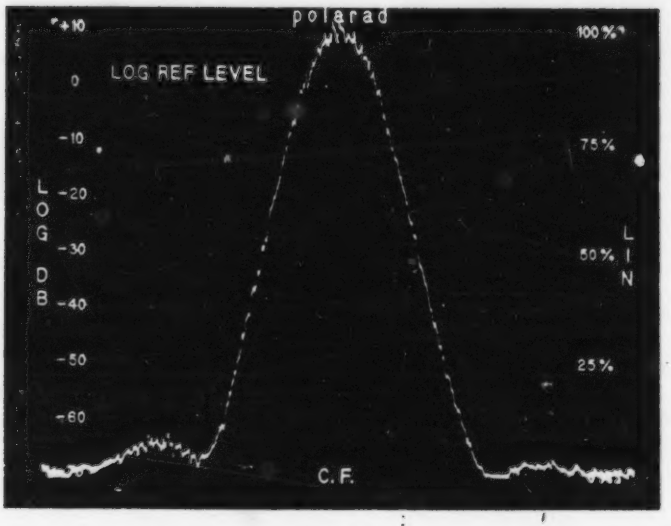

Fig. 1: Spectrum of Back Reflection (Transducer perfectly aligned) Centre Frequency 32 MHz.

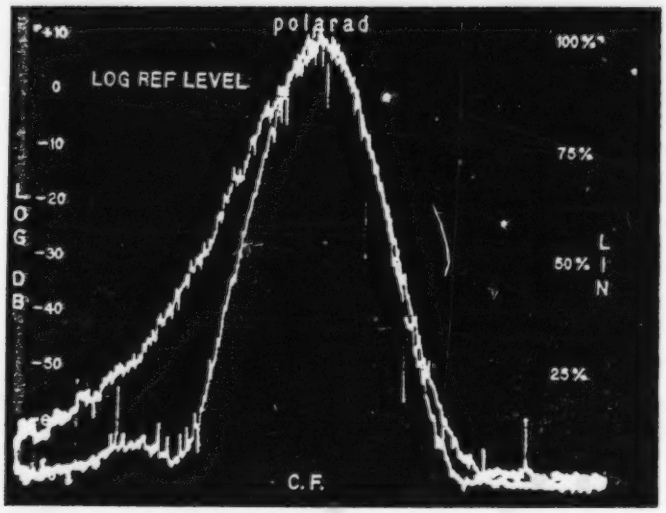

Fig. 2: Spectrum of Back Reflection (Transducer misaligned, compared with properly aligned spectrum)

For analysis of the defect signal, two types of defects were categorized, i.e., bubbles and inclusions. For analysis of the ultrasonic signal itself, it was found that standardization was necessary. This was accomplished as described under "Standardization" by spectrum analysis of the signal reflected from the bottom of the glass sample. A perfect 90° reflection is expected to give rise to a Gaussian frequency distribution about the central frequency. Figure 1 shows the near-perfect spectrum used for standardization. Tilting the transducer to any other angle along directions permitted by the rig, increases the intensity of the side lobe relative to the main frequency (Fig. 2). The standardization procedure provided a uniformity of signals to the point that bubble signals could be considered comparable and signal size was directly related to the size of the defect. Fig. 3 shows a normalized diagram of a number of reflections of large bubbles; note the nearly perfect relationship.

Previously reported correlations at 14 and 25 MHz showed similar relationships but were much less accurate since the standardization procedure was not used. For bubble reflections smaller than the focal diameter of the ultrasonic beam a certain spread is observed (Fig. 4) but the signals are basically similar to the reference back reflection (note the rectangles which indicate the spread of the back reflection signals).

Plotting bubble signals using the "key" of the reflection (the second negative peak) which is also the maximum signal, the relationship with bubble size is shown in Fig. 5. Figure 6 shows the same data but emphasizes the small defects. Here the smallest defect analyzed is $\sim$ 50 µm ø.

With inclusions,the analysis is more complex. Signal shapes are different from those of bubbles although sometimes similarities are observed. Most similar-looking signals appear to be upsidedown compared with bubble signals. A bubble signal shows one glitch going up, before the signal proper starts, and with inclusions it sometimes takes two or three glitches before the signal starts (Fig. 7). Of the 23 inclusions investigated (some tested from both sides) 2 signals indicated bubbles (Fig. 8). Microscopic analysis did indicate a bubble-shaped defect but with an opaque inclusion within it. As the reflection indicated an acoustic impedance lower than that of glass, the inclusion was probably loose in the bubble. The total tally of inclusion defects is shown in Table I:

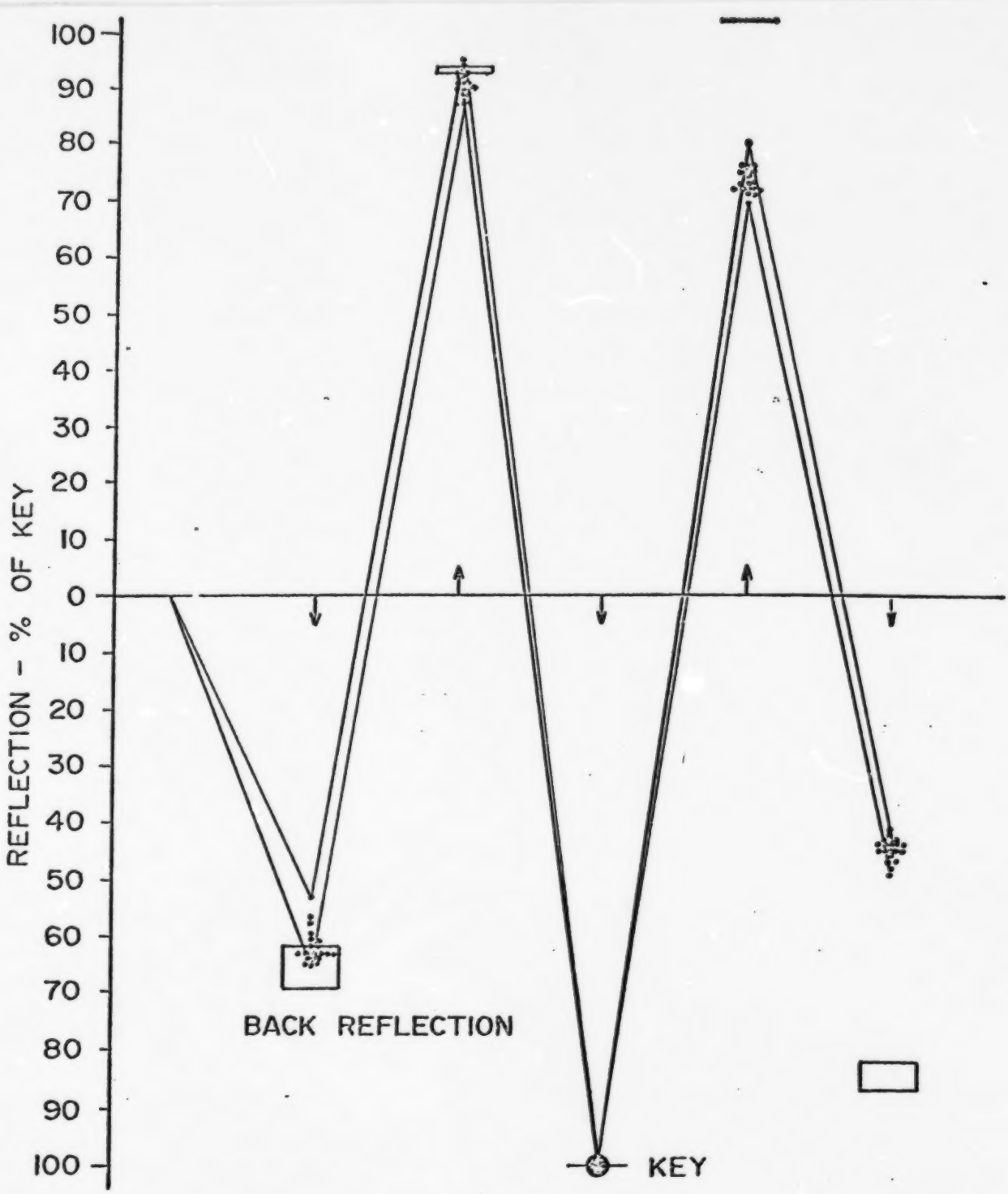

Fig. 3: Reflections at 32 MHz

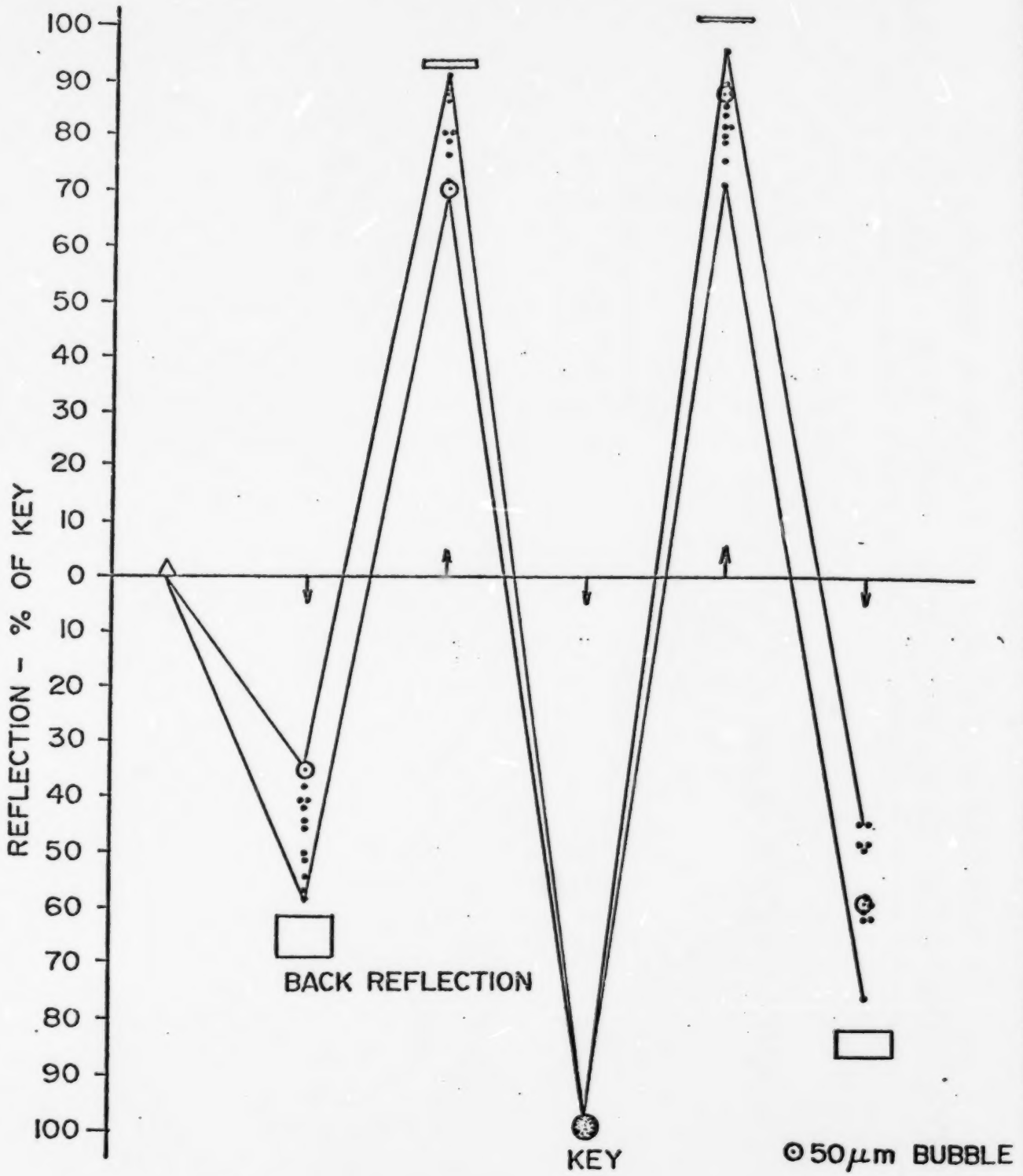

Fig. 4: Reflections at 32 MHz

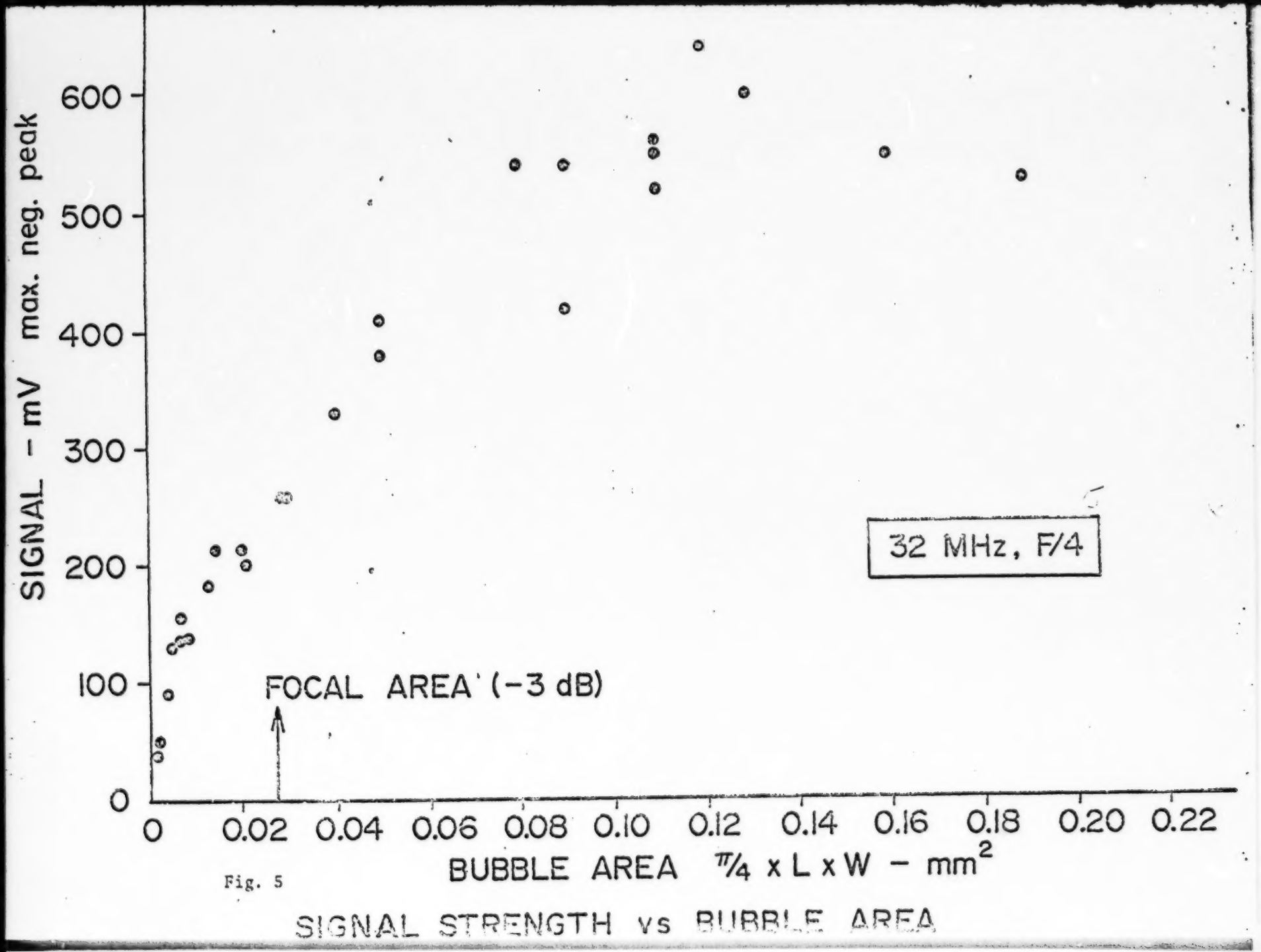

Fig. 5

SIGNAL STRENGTH vs BUBBLE AREA

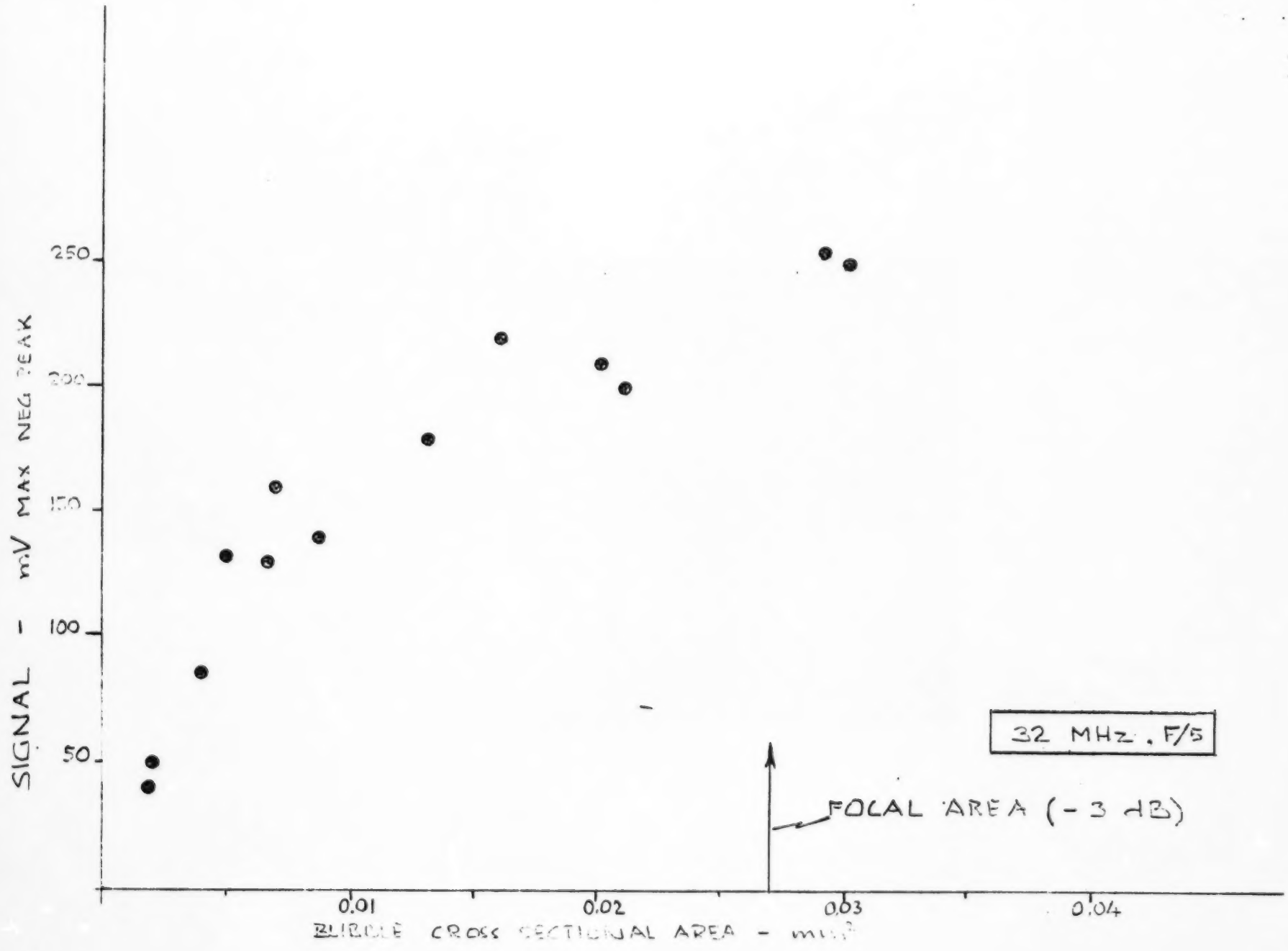

FIG 6 SIGNAL STRENGTH vs BUBBLE AREA

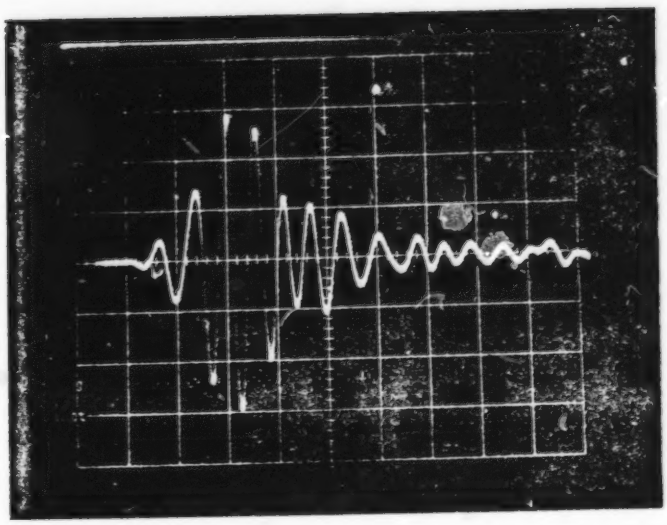

Fig. 7: Inclusion Reflection

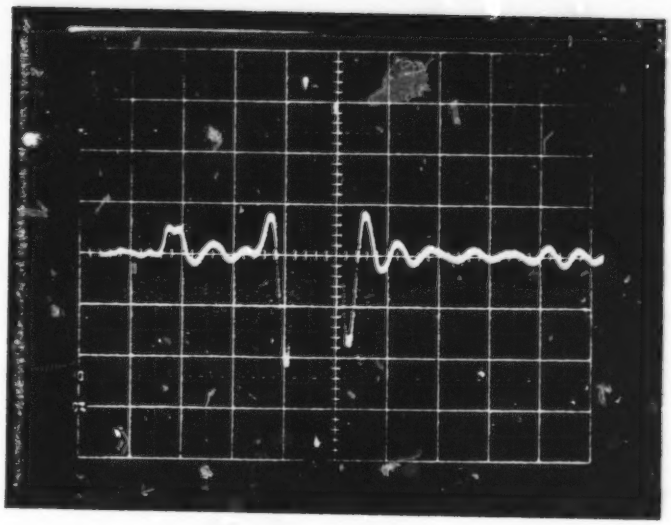

Fig. 8: Inclusion which showed as bubble

TABLE I, 23 Inclusions (some tested from both sides)

Long Signals	8	(Fig. 9)
Upside-down signals	9	(Fig. 10)
Multiple-glitch signals	6	(Fig. 7)
Bubble signals*	2	(Fig. 8)

*one sample

It is probable that detailed inclusion analysis will come eventually from the signal spectra as inclusions show marked spectral changes (Fig. 11 and 12). At present, only hypothetical relationships have been put forward; the real analysis will follow later.

Studies in Ceramics

In addition to the work done on visible glass defects, tests of ceramic ionic conductors have been performed. The objective of these investigations was to discover if ultrasonics could be used to check the integrity of the ceramics before and after ion exchange, a process which potentially causes cracking within the sample. Cracks are often initiated at small inclusions which did not sinter properly and these particles should be detectable before the ion exchange is performed.

Initial studies were performed on samples which had already been ion-exchanged. The oscilloscope trace of the reflected signals from a 2.5 mm thick sample displayed reflections from the top and bottom surfaces and a large amount of scattering within the sample believed related to the grains. However, no strongly reflecting surface was located. A 6.3 mm thick sample was also investigated for which no reflection from the bottom of the sample was found; but some surfaces inside the sample returned signals strong enough to indicate on the recording equipment (Fig. 13). After cutting the sample vertically through the area indicated, no crack could be seen using the optical microscope (Fig. 14), but examination by scanning electron microscopy revealed extensive cracking (Fig. 15) and a void, possibly associated with a remnant SF/FD sphere.

Subsequently, tests were performed on a before-and-after ion-exchange basis and no signals of unsintered particles in the pre-exchange samples were of sufficient intensity to be recorded. After ion-exchange, however, some samples did display evidence of cracking.

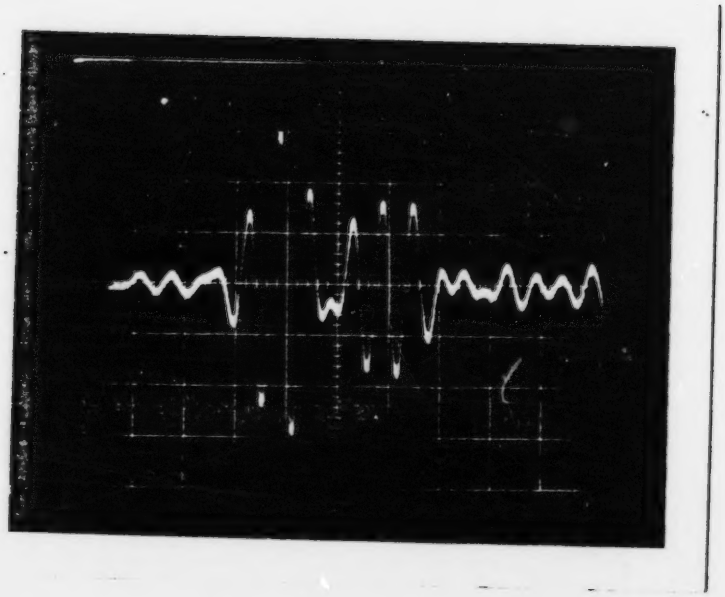

Fig. 9: Long Inclusion Signal

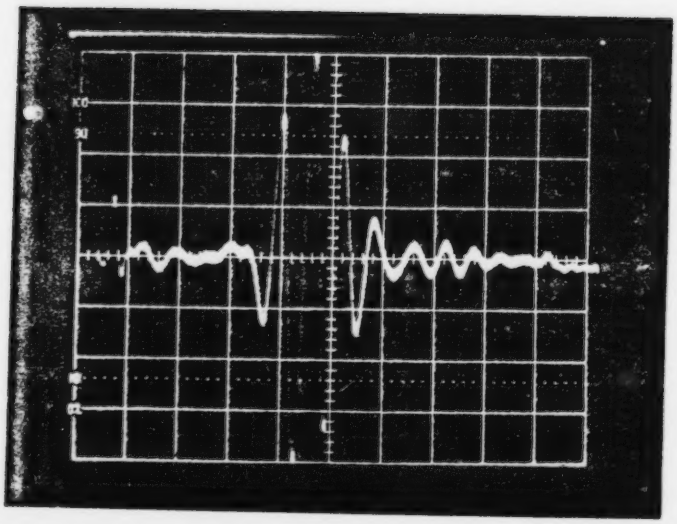

Fig. 10: Upside-Down Inclusion Signal

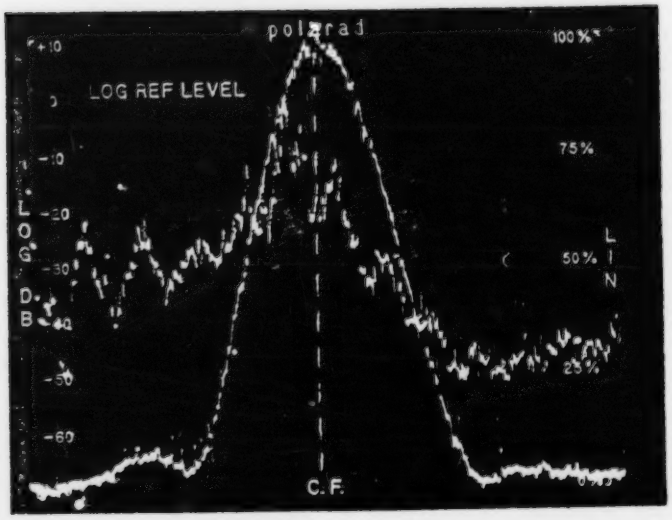

Fig. 11: Spectrum of Small Inclusion

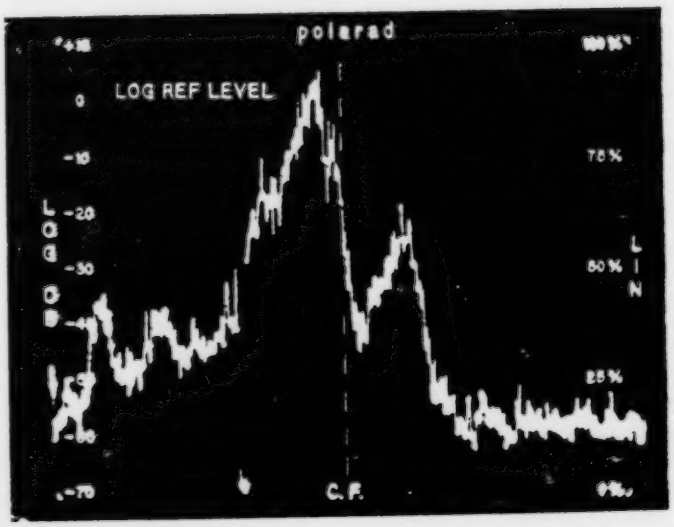

Fig. 12: Signal of Large Inclusion

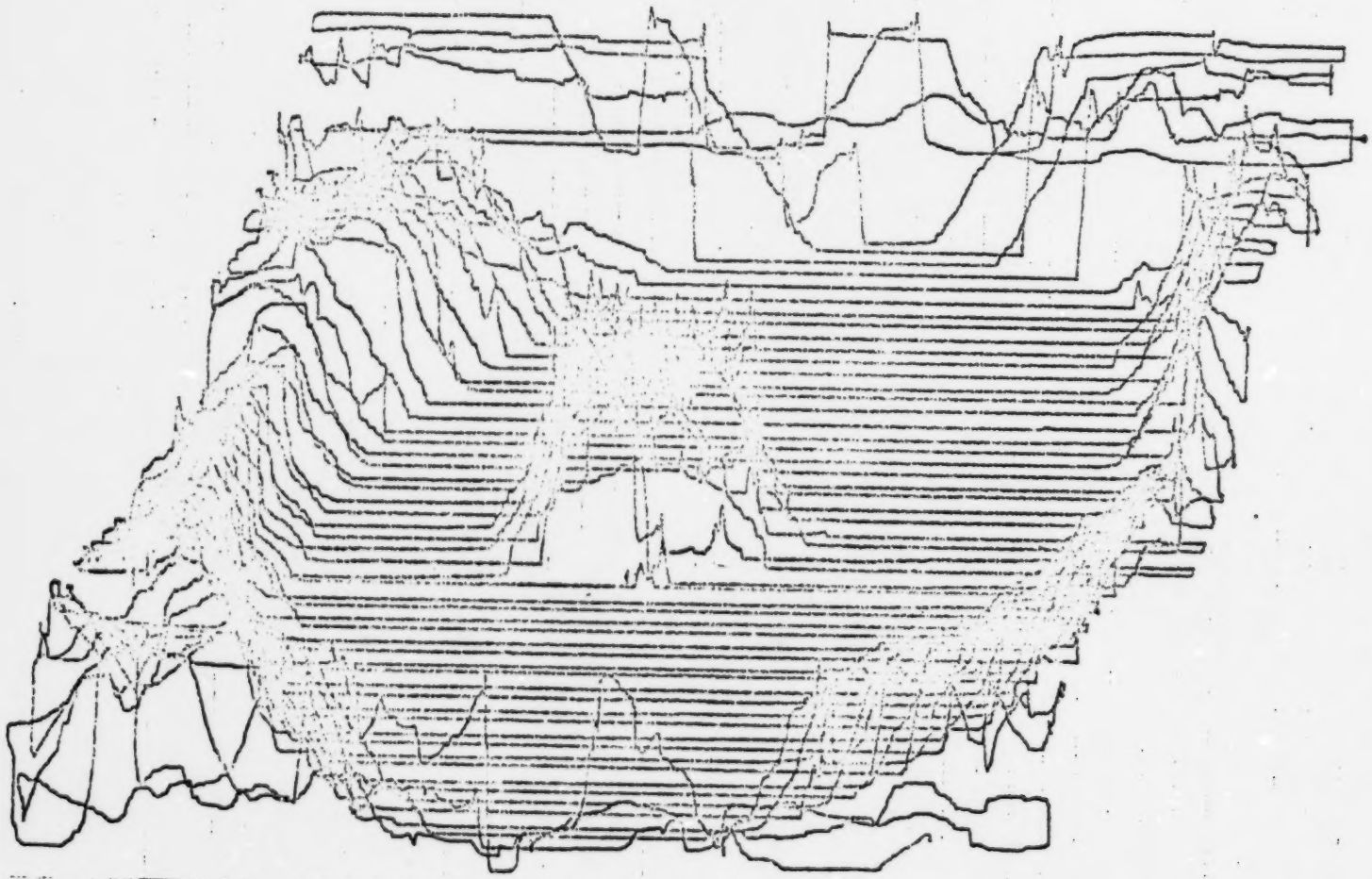

FIG 13 ULTRASONIC SCAN OF 1st LAYER

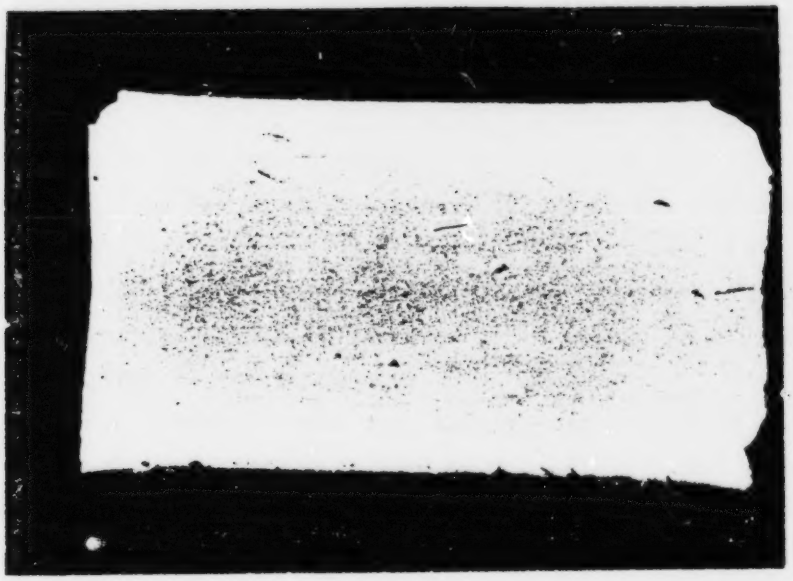

Figure 14

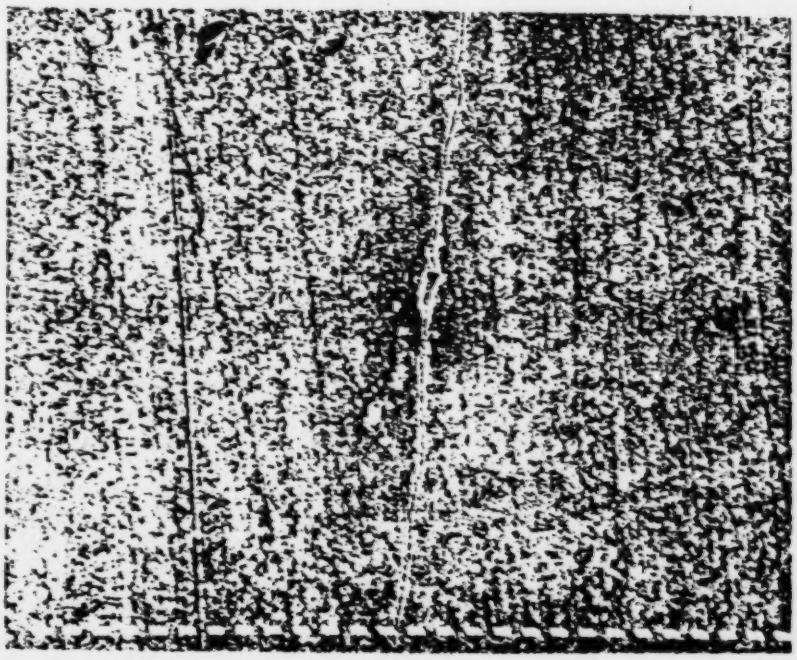

Figure 15

Transducer Development

Because of the high sound velocity in ceramics and the need for extremely high sensitivity, the required ultrasonic transducer must have an extended, small-diameter focus; such transducers do not exist. However, two developments, both in Canada, may make it possible to build a satisfactory high frequency transducer. The first is a facility at Queens University (2) where Dr. Sayer conducts experiments depositing very thin layers of piezo-electric substances on a prepared base. The second is the development of an extended focus transducer by R.V. Murphy. (3,4) He has shown by experiment that it is possible to obtain an extended focus much longer than with conventional transducers.

The transducers must be of a very high frequency (100 MHz) with micro- or screwdriver-blade-focus to penetrate the ceramic material and yet to cover a fair volume of material at each pass. In the development programme, thin piezo-electric discs have been ground to approximately 0.1 mm and then poled for ultrasonic use. This stage was added to learn about poling piezo-electric crystals.

Parallel to this development, was the depositing at Queens University of piezo-electric layers on previously-prepared substrates. In this development, the first round has been completed and improvements proposed; this has led to a change in operating sequence which will produce a working transducer soon after deposition of the sensitive layer.

In the grinding of flat discs it was found that the machinery was too coarse to produce discs < 0.1 mm. Several thinned discs contained defects which caused an electrical short on poling. Other discs simply did not have the quality to sustain the recommended 600 V/0.11 mA requisite for poling 0.1 mm thickness.

In spite of these problems, alot has been learned about the manufacture of high frequency transducers. Articles and other information have been gathered and the future attempts should be facilitated.

Scanning Facility

The scanning facility should consist of the scanner itself, precisely controlled and of high quality, and a signal processing facility. Ideally, all flaws spotted should be measured and listed automatically by computer. Such a scanner is currently under design. It is planned to move a transducer in the x-y plane and allow any container to be used. If in the future round or

tubular parts have to be scanned, such a facility could be added without making the scanner obsolete.

Flaw signals have to be "extended" before they can be processed by the computer (converted from their momentary appearance to dc. which lasts long enough to register in the computer memory or to move a pen on an x-y recorder). This facility has been built and is operational; it works in conjunction with the Panametrics pulser receiver unit but still lacks definition because of the inadequate rejection of spikes outside the gate of the pulser receiver.

The scanner was originally controlled from a TRS 80 computer which handled the scanning. The scanning programme has been transferred to a Digital PDP 11/23 computer and this machine will also be programmed for memorizing the flaws and their position. In this way, the computer will return to a previously-recorded flaw and allow further analysis to be made. This feature is extremely important as the flaws are so small as to be impossible to relocate by manual manipulation of the scanner.

The addition of the graphics board to the computer has eliminated the x-y recorder as the major flaw recorder. The computer programme is not yet complete enough to perform all the functions described above but it is gradually being modified to perform these functions.

Conclusions

Over the past year, a thorough understanding has been reached of the problems related to the ultrasonic detection of small flaws and cracking in super-ceramics and ceramic ionic conductors.

Equipment has been obtained to assist in the research programme.

Equipment improvement and the addition of a computer has improved the capability of locating and analyzing small flaws.

The relationship between signal and bubble size has been empirically established for bubbles in glass.

Analysis of the spectra from glass inclusions is underway.

References

1. Schlengermann, The Characterization of Focussing Ultrasonic Transducers by means of Single Frequency Analysis Materials Evaluation, December 1980, p.73.

2. Magnetron Sputtering of PZT and ZnO films by M. Sayer, Queens University, Kingston, Ontario.

3. R.V. Murphy, Focussed Ultrasonic Probes for Contact Inspection, Materials Evaluation, September 1980, p.53.

4. R.V. Murphy, Toroidal, Conical and Spherical Lenses in Ultrasonic Inspection, Materials Evaluation, March 1981, p.391.